STUDIES OF THE MICROMECHANISMS OF THE FLOW AND FRACTURE OF ICE

FINAL REPORT

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13. ABSTRACT *(Maximum 200 words)*

The goal of this work was to understand the behavior of dislocations around loaded notches in ice single crystals using x-ray topography and to study the effects of both orientation and impurities on the flow of ice single crystals.

Ice single crystals were grown in Dartmouth's Ice Research Laboratory. For dislocation observations, stress-controlled in-situ deformation experiments were performed using an x-ray beam line at the National Synchrotron Light Source in collaboration with Dr. D. Black and Prof. M. Dudley. The observed dislocation motion around notches of different orientations was also modeled.

Compression tests on high-purity ice single crystals of different orientation showed that Schmid's critical resolved shear stress law is well-obeyed in ice.

It was also shown for the first time that a few p.p.m. of sulfuric acid markedly reduced both the peak strength and subsequent flow stress of ice single crystals at -20°C, and increased their ductility.

Measurements of the chemistry of triple points and nodes in both laboratory-grown, sulfuric acid-doped ice and ice cores from Antarctica using x-ray microanalysis in a SEM have shown strong segregation in the former ice but little in the latter.

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INTRODUCTION

Work funded by ARO (Grant No. DAA-H04-96-1-0041) was initiated on April 1st, 1996, under the direction of the author on "Studies of the Micromechanisms of the Flow and Fracture of Ice". The long-term goals of this work are to: (1) provide the microstructural basis for modeling the plastic flow and fracture of ice; and (2) understand how impurities change the mechanical properties of ice. The near-term goals of this project were to: (1) examine the behavior of dislocations around notches in single crystal ice of different orientations loaded at different temperatures; (2) determine the effect of crystal orientation on the strength of ice; and (3) determine the effect of sulfuric acid, the most common impurity in ice in central Antarctica and Greenland, on both mechanical behavior and dislocation behavior in ice.

DISLOCATION BEHAVIOR AROUND LOADED NOTCHES

Dislocation behavior around notches in ice single crystals, grown from purified, de-gassed water, has been studied by *in-situ* deformation synchrotron x-ray topography. Two millimeter thick specimens were transported to the National Synchrotron Light Source at Brookhaven National Laboratory (BNL), New York, USA, and transferred to a testing jig. The jig can apply loads, in tension or compression, up to 445 N, corresponding to stresses up to 20 MPa. X-ray topographs were obtained by allowing a highly-collimated beam (5 mm high x 10 mm wide) of white X-rays to impinge on the specimen during loading. Several diffraction spots were recorded simultaneously in a single exposure, which typically took ~2s. Each Laue diffraction spot is an image of the grain in which both grain boundaries and dislocations can be observed.

The dislocation structures around notches in ice single crystals of several orientations loaded under compression at a variety of temperatures have been studied. The behavior of dislocations in the region around loaded notches in ice single crystals has also been investigated using a computer model. The model predicts a dislocation mobility (unit distance/(unit time x unit stress)) approximately 4.5 times slower than that found by Shearwood and Whitworth [Shearwood, C. and Whitworth, R., *The Velocity of Dislocations in Crystals of HCl-Doped Ice*, Phil. Mag., 65, 1, 1992, 85-89.]. As suggested by Dr. David Cole of USA-CRREL, this difference could be due to localized stress inhomogeneities inflating the Shearwood and Whitworth results. Interestingly, no dislocation free Zone (DFZ) was observed ahead of the notch. Experimentally it was observed that the interaction of the stress field around the notch led to a dislocation-depleted zone (DDZ) on one side of the notch (where dislocations had completely run out of the specimen) and the formation of a low angle boundary on the other side of the notch. This was well-described by the computer model as the expansion of basal plane dislocation loops

on the DDZ side of the notch and the shrinking of similar dislocation loops on the low angle boundary side of the notch. The lack of a DFZ ahead of loaded notches has implications for modeling the fracture of ice.

THE EFFECT OF ORIENTATION ON THE STRENGTH OF ICE SINGLE CRYSTALS

Compression tests were performed on ice single crystals as a function of orientation at an axial strain rate, $\dot{\varepsilon}$, of 1 x 10^{-5} s^{-1} at -20°C. These clearly showed that Schmid's law is obeyed, i.e. the resolved (peak) shear stress does not depend on orientation, see Figure 1. To analyze the experimental data, it was necessary to determine the stress exponent, n, for ice single crystals in the relationship $\dot{\varepsilon} = A\sigma^n$, where A is a constant and σ is the stress, and to compensate for the different shear strain rates on the basal plane experienced by crystals of different orientations. At –20°C, n was found to be ~ 1.95, a similar value to those obtained by others on single crystal ice. Our observation that Schmid's law is obeyed, i.e. that there is no orientation dependence of the critical resolved shear stress, indicates that hydrostatic stresses play no part in dislocation motion in ice, and that it is valid to resolve shear stresses onto the basal slip system using this law. This latter feature has previously been assumed by many workers but was unproven.

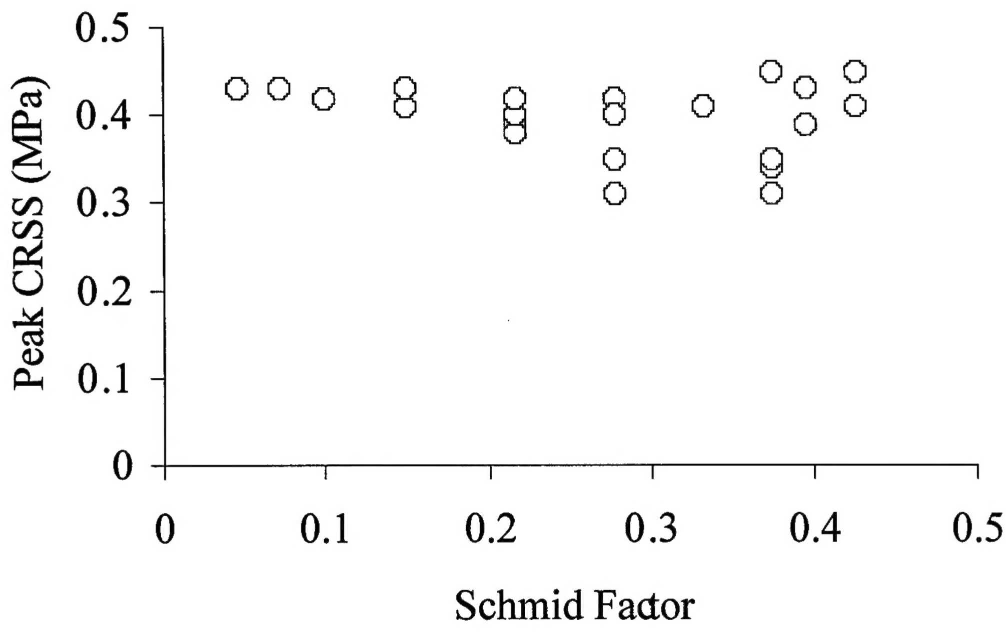

Figure 1. Peak critical resolved shear stress, CRSS, (from 3° to 70°) normalized to a basal slip plane shear strain rate of 1×10^{-5} s^{-1}, at –20°C versus the Schmid factor (Cos σ Cos λ).

THE EFFECT OF H_2SO_4 ON THE STRENGTH OF ICE SINGLE CRYSTALS

Compression tests were performed at a variety of axial strain rates on ice crystals of fixed orientation doped with H_2SO_4 at -20°C. These showed that the stress exponent is 1.95 for sulfuric acid-doped ice, i.e. the same as for undoped ice.

Mechanical tests performed on ice single crystals of fixed orientation but different sulfuric acid concentrations at 1×10^{-5} s^{-1} at -20°C showed that this dopant dramatically decreases both the peak stress and the subsequent flow stress, see Figure 2. Similar behavior has been noted previously for HCl and HF doping of similar levels at -70°C (S.J. Jones, Phys. Lett., 25A (1967) 366; S.J. Jones and J.W. Glen, Phil. Mag., 19 (1969) 13). However, this is the first time such an effect has been observed from this naturally occurring dopant.

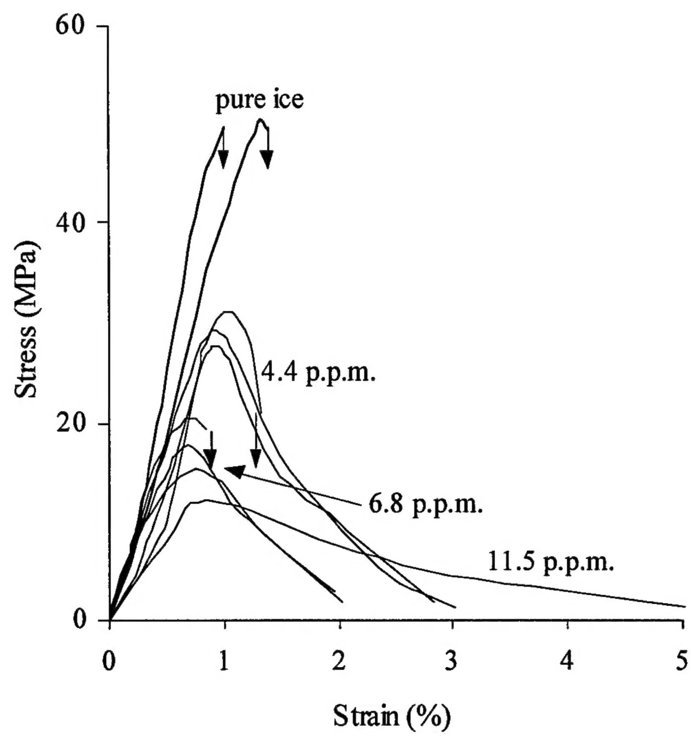

Figure 2. Comparison between engineering stress-strain curves for sulfuric acid-doped and undoped ice single crystals with the C axis at 3° to the loading direction, at an initial axial strain rate of 1×10^{-5} s^{-1} at -20°C. The downward arrows indicate specimen failure, which occurs at higher strains with increasing sulfuric acid concentration.

It was found that the peak stress decreased with the square root of the sulfuric acid concentration, see Figure 3, a similar dependence to the solute *hardening* encountered in metals.

Interestingly, the presence of sulfuric acid also produces greater ductility, see Figure 2. This latter feature could simply be related to the lower peak stress and flow stress. Examination of sulfuric acid-doped single crystals by x-ray topography indicated that this dopant dramatically increases the grown-in dislocation density.

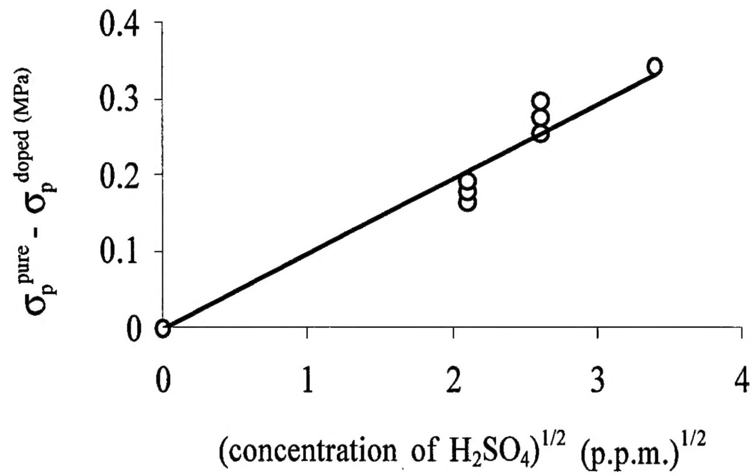

Figure 3. Graph of the difference in CRSS (peak stress) between undoped ice and sulfuric acid-doped ice crystals ($\sigma_p^{pure} - \sigma_p^{doped}$), for a loading direction 3° to the C axis, versus the square root of the concentration of sulfuric acid for crystals compressed at –20°C, normalized to a basal shear strain rate of 1×10^{-5} s^{-1}.

THE STRUCTURE AND CHEMISTRY OF POLYCRYSTALLINE ICE

Specimens were made from Greenland Ice Sheet Project (GISP2) core samples obtained from depths 94, 214, 341, 2750, 2850, and 2950 m below the surface and from Byrd Station core samples obtained from depths 101, 341, 1892, 1992, and 2090 m below the surface. X-ray topographs revealed dislocation densities, which varied from low (1×10^{-7} m^{-2}) to unresolvably high. For specimens above 341 m the dislocation density increased as depth increased until recrystallized grains began appearing, at which point both high and low dislocation density grains could be observed at the same depth. For the specimens below 1892 m the grain size markedly increased as depth increased. The deepest specimens from both the GISP2 core and the Byrd Station core were composed almost entirely of a single crystal for our specimen size of 2 mm x 10 mm by 25 mm. All the grains from specimens below 1892 m were highly distorted. The only other topographs obtained from glacier ice have been by Fukuda and Shoji (Fukuda, A. and H. Shoji, 1988, *Lattice defects in ice crystals, x-ray topographic observations*, A. Higashi, ed.,

Sapporo, Japan: Hokkaido University Press, p13) who imaged ice taken from both the surface of the Mendenhall Glacier and from Byrd Station deep core ice (2000 m in depth). In the deep core ice, they observed a mosaic structure. Our topographs indicate no evidence of a mosaic structure, but only highly distorted grains.

In addition to the X-ray topographs, preliminary observations have been performed on the ice core specimens using a scanning electron microscope (SEM) equipped with an energy-dispersive x-ray microanalysis system (EDS). (The SEM/EDS were both acquired using funds from ARO.) Grain boundaries and grain triple junctions have been observed using the SEM, and triple junction and node chemistry (particularly sulfate presence) was measured using the EDS system. The overall specimen chemistry was determined using ion chromotography.

Personnel Supported

During this period the following personnel were wholly or partially supported by the grant:

D. Cullen - Ph.D. candidate
Y.L. Trickett - M.S. candidate
P.M.S. Pradhan - undergraduate assistant
I. Baker - P.I.

Y.L. Trickett completed her M.S. in 1999 on "X-ray topography of Doped Ice" with partial support from ARO. Dr. David Cole of USA-CRREL was her external examiner. For his work on ice, P.M.S. Pradhan won Thayer School's Francis L. Town prize for 1999-2000 for the most outstanding Engineering Science undergraduate. D. Cullen aims to finish his Ph.D. thesis in summer 2000. Dr. David Cole will be his external examiner.

FUTURE WORK

In future, we will extend our work to the study of microstructures and deformation conditions that are more realistic of ice in nature. We will study quantitatively the dislocation behavior associated with flow and fracture under a variety of conditions. In particular, we will study:

1) The relationship between dislocation density and the flow stress as a function of temperature;
2) The relationship between the apparent or effective elastic modulus and the dislocation density;
3) Flow and fracture during high-temperature deformation involving grain boundary sliding.

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SUMMARY

In summary, our x-ray topography work provides a fundamental micromechanistic basis for modeling the mechanical properties of ice. We have also recently shown that sulfuric acid, the most important impurity in ice sheets, has dramatic effects on the mechanical properties of ice, a result which is important for modeling the effect of climate change on the flow of ice sheets. We have also shown that Schmid's law is obeyed in ice.

The addition of an x-ray microanalysis facility to our environmental scanning electron microscope, coupled with our use of x-ray topography means that we can now characterize polycrystalline ice in terms of grain-size, crystalline defects and chemistry (both local and overall).

PRESENTATIONS

1. "Dislocation in Ice", I. Baker, University of Wisconsin (1996).

2. "Dislocation in Ice", I. Baker, Oak Ridge National Laboratory (1997).

3. "Dislocation in Ice", I. Baker, Rensselaer Polytechnic Institute (1997).

7. "Dislocation in Ice", I. Baker, University of Sydney, Australia (1997).

8. "Synchrotron X-ray Topographic Studies of Dislocations in Ice" I. Baker, X. Hu, D. Cullen, X. Li, M. Dudley, and D. Black, Annual TMS Meeting, San Antonio, TX, February, 1998.

9. "Dislocation in Ice", I. Baker, Climate Change Research Center, University of New Hampshire (1999).

10. "Dislocation in Ice", I. Baker, U.S. Army Cold Regions Research and Engineering Laboratories (1999).

11. "The Mechanical Properties of Ice Single Crystals" Y. L. Trickett and I. Baker, poster presented at the Eighth Annual Midwest Glaciology Meeting, Boulder, CO, March 15-16, 1999.

12. "Synchrotron X-ray Topographic Observations of the Interactions of Dislocations with Grain Boundaries in Ice", I. Baker, F. Liu, K. Jia, X. Hu, D. Cullen, D. Black and M. Dudley, poster at the American Geophysical Union Spring Meeting, May 31-June 4, 1999.

13. The Mechanical Properties of Ice Single Crystals", I. Baker, Y.L. Trickett and P.M.S. Pradhan, poster at the American Geophysical Union Spring Meeting, May 31-June 4, 1999.

14. "X-ray Topographic Observations of Dislocation/Grain Boundary Interactions in Ice", I. Baker, F. Liu, K. Jia, X. Hu, D. Cullen and M. Dudley, poster at the International Symposium on the Verification of Cryospheric Models: bringing data and modeling scientists together, Zurich, Switzerland, 16-20 August, 1999.

15. "Deformation of Ice Single Crystals with and without H_2SO_4", I. Baker, Y.L. Trickett and P.M.S. Pradhan, poster at the International Symposium on the Verification of Cryospheric Models: bringing data and modeling scientists together, Zurich, Switzerland, 16-20 August, 1999.

16. "The Effect of H_2SO_4 on the Stress Exponent in Ice Single Crystals", I. Baker, Y.L. Trickett and P.M.S. Pradhan, International Conference on the Deformation of Glacial Materials, London, United Kingdom, September 6-8, 1999.

17. "The Strength of High-Purity and Acid-Doped Ice Crystals", Y.L. Trickett P.M.S. Pradhan and I. Baker, poster at the Fall TMS meeting, Cincinnati, OH, Oct 1-4, 1999.

18. "The Effect of Orientation and sulfuric Acid on Ice Single Crystals", U.S. Army Cold Regions Research and Engineering Laboratories (2000).

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1. "Dynamic In-situ Synchrotron X-ray Topographic Observations of Dislocations in Notched Ice Crystals", X. Hu, I. Baker and M. Dudley, in "Applications of Synchrotron Radiation to Materials Science", L. Terminello, S. Mini, D. L. Perry, and H. Ade (Eds.), Mat. Res. Soc. Symp. Proc., 437, (1996) p119-124.

2. "Temperature Dependence of Dislocations in Notched Ice", X. Hu, I. Baker and M. Dudley, Journal of Physical Chemistry B, **101** (1997) 6102-6104.

3. "Observation of Dislocations in Ice", I. Baker, Journal of Physical Chemistry B, **101** (1997) 6158-6162.

4. "Dislocation Motion at Notch-Tips in Ice Single Crystals: Experiments and Interpretation", D. Cullen, X. Hu, I. Baker and M. Dudley, Cold Regions Science and Engineering, in press.

5. "The Orientation Dependence of the Strength of Ice Single Crystals", Y. L. Trickett, I. Baker and P.M.S. Pradhan, Journal of Glaciology, in press.

6. "The Effects of Sulfuric Acid on the Mechanical Properties of Ice Single Crystals", Y. L. Trickett, I. Baker and P.M.S. Pradhan, Journal of Glaciology, in press.

7. "The Effect of H_2SO_4 on the stress exponent in Ice Single Crystals", I. Baker, Y.L. Trickett and P.M.S. Pradhan, Annals of Glaciology, in press.

8. "X-ray Topographic Observations of Dislocation/Grain Boundary Interactions in Ice", I. Baker, F. Liu, K. Jia, X. Hu, D. Cullen and M. Dudley, Annals of Glaciology, in press.

9. "Dislocation Motion Around Loaded Notches in Ice Single Crystals", D. Cullen, X. Hu, I. Baker and M. Dudley, Proceedings of the Material Research Society, in press.

10. "Preliminary Synchrotron X-ray Topographic Images of Ice from the Greenland Ice Sheet Project (GISP2)", D. Cullen and I. Baker, submitted to Antarctic Journal: Review 1998.